Vacuum 102 (2014) 5–11

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Effects of the surface oxide layer on platelet growth in H_2^+-implanted Si

B.S. Li*, Z.G. Wang

Institute of Modern Physics, Chinese Academy of Sciences, Lanzhou 730000, PR China

ARTICLE INFO

Article history:
Received 17 July 2013
Received in revised form
27 September 2013
Accepted 31 October 2013

Keywords:
Hydrogen implantation
Platelet
Microcrack
Annealing
Smart-cut

ABSTRACT

The effect of a surface oxide layer on platelet growth in H_2^+-implanted Si was investigated. Samples of p-type Cz Si (100) and the same Si covered with a 170 nm thick thermal oxide layer were implanted with H_2^+ ions to a fluence of 2.5×10^{16} H_2^+/cm^2 at room temperature. Post-implantation thermal annealing at temperatures between 773 K and 973 K for 30 min was performed in a flow of N_2. Optical microscopy, cross-sectional transmission electron microscopy (XTEM) and micro-Raman spectroscopy were performed to investigate the effect of the oxide layer on platelet evolution upon annealing. Optical microscopy observations show that blisters and half-open blisters occur on the surface of Si with the oxide layer, but craters and half-open blisters occur on the pure Si surface. XTEM observations show that the growth rate of platelets in the defect band of Si with the oxide layer is slower than that in the pure Si sample, due to the lower concentration of vacancy-type defects in the Si with the oxide layer. The density of frank loops increases with increasing annealing temperature in the Si with the oxide layer.

1. Introduction

Bruel firstly reported the fabrication of silicon on insulator (SOI) materials by hydrogen implantation in silicon, called "Smart-Cut" technology [1–9]. The procedure of this technology comprises hydrogen implantation in silicon, then bonding them to a substrate stiffener. Finally, a splitting treatment is used to achieve layer transfer upon annealing. In detail, H implantation-induced kinds of types of Si–H complexes in the damaged layer, including two-dimensional vacancy-type defects named platelets. After annealing at a temperature of approximately 773 K, microcracks are formed in the region of maximum damage due to the growth of platelets via Oswald ripening [10]. Those microcracks are parallel to the sample surface and therefore, a full layer is transferred from the parent substrate [4,5]. However, in the case of nonbonded implanted wafers, blisters and craters are formed on the surface [3,8,9]. Because the activation energy is the same between the start layer splitting and blister formation [11], the growth of blisters and craters in hydrogen implanted silicon have attracted more attention.

In order to fabricate SOI materials, the differences of the electrical insulation material SiO_2 formed on the stiffener surface and on the parent wafer surface before hydrogen implantation are still unclear. Therefore, it is crucial to elucidate whether the presence of surface oxide layer can affect the platelet growth in hydrogen implanted silicon. The aim of this paper is to investigate the influence of the top oxide layer on the evolution of H-induced platelets in silicon.

2. Experimental

Two sets of Czochralski grown p-type Si (100) with resistivity of 5–8 Ω cm were used. One set Si sample was covered with a 170 nm thick thermal oxide layer. The oxide layer was grown on Si by thermal oxidation processes at 1373 K in wet oxygen ambient for 70 min. For comparison, the other set Si sample is the same Si bulk without an oxide layer on the surface. These samples were implanted at room temperature with 100 keV H_2^+ ions to a fluence of $2.5 \times 10^{16}/cm^2$. The beam density was kept at 0.7 μA/cm^2. The experiment was finished at the 320 kV platform for multi-discipline research with highly charged ions at the Institute of Modern Physics, Chinese Academy of Sciences. Due to the density of SiO_2 as the near same as that of the pure Si, the presence of the SiO_2 has no significant influence in the mean projected ranges (R_p). According to the TRIM simulation [12], the R_p value for 100 keV H_2^+ ions in a Si sample is about 560 nm. Post-implantation, the samples were cut into several pieces. Some of the pieces were subjected to furnace isochronal annealing at temperatures of 773 K and 973 K in a flow of N_2 ambient for 30 min.

Olympus optical microscopy was used to study the surface morphology properties of the samples. The magnification was

* Corresponding author.
E-mail address: b.s.li@impcas.ac.cn (B.S. Li).

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http://dx.doi.org/10.1016/j.vacuum.2013.10.027

1000×. Before observations, the samples were cleaned in ethanol by ultrasonic for 5 min. Cross-sectional transmission electron microscopy (XTEM) was carried out to reveal the defect microstructure. Before XTEM observations, the samples were cut, glued and then thinned using mechanical polishing and final Ar-ion milling at room temperature. XTEM images were taken at 200 kV with a JEOL 2010 microscopy. The samples were observed near the [011] direction. The micrograph conditions were bright field (BF) and weak-beam dark field (WBDF) with the image condition: (g, 5 g), $g = 111$. The distribution of defects under WBDF was counted. The thickness for observation of defect was determined by thickness fringes with an uncertainty of about ±15%. In addition, in order to research the evolution of chemical bonds, micro-Raman spectroscopy (μ-RS) was used. μ-RS was performed using a Horiba Jobin Yvon HR-800 spectrometer. The excitation wavelength was 514.5 nm from the Ar^+ laser and the power was 5.5 mW. The laser spot was about 1 μm, and the linewidths have a spectral resolution better than 0.42 cm^{-1}.

3. Results

3.1. Surface morphology measurements

Compared to unimplanted Si wafers, the surfaces have no any changes for the as-implanted samples, as shown in Fig. 1(a) and (d). After annealing at 773 K, the sample surfaces have significant changes and the surface morphologies are different between the Si with the oxide layer and pure Si sample. Fig. 1(b) presents that dense blisters and few half-open blisters are displayed on the surface of Si with the oxide layer. However, craters, half-open blisters and very few blisters are exhibited on the surface of pure Si sample, as shown in Fig. 1(e). The densities of craters and half-open blisters are similar. With increasing temperature up to 973 K, the density of half-open blisters increases while the density of blisters decreases, indicating coalescence of blisters during the thermal annealing. Very few craters are shown on the surface of Si with the oxide layer, as shown in Fig. 1(c). For comparison, there are growing numbers of craters on the surface of pure Si sample, as shown in Fig. 1(f).

3.2. XTEM analysis of the damage layer

To achieve more information about the hydrogen implantation-induced damage evolution upon annealing, XTEM imaging of the sample in the annealing state was performed. After annealing at 773 K for 30 min, Fig. 2(a) presents a XTEM image of the implanted region in the Si with the oxide layer. The image exhibits a well-defined damage band at depths ranging from 500 nm to 620 nm as compared to a well-defined damage band at depths ranging from 430 nm to 620 nm in the pure Si (Fig. 2(b)). The high magnification shows the damage band that is mainly made up of cavities and dislocation loops in Fig. 3(a). These cavities are in a penny shape named platelets, which are distributed all over the defect band. The platelets have diameters ranging from 3 nm to 28 nm with a mean value of about 11 nm, and about 1 nm in thickness. Moreover, most of platelets are aligned parallel to the sample surface in the (100) plane, located all over the damage band. In addition, some {111} platelets are found in the periphery of the damage band. However, the microstructures in the pure Si are different from those of the Si with the oxide layer [see Fig. 3(c)]. A significant microcrack was formed in the damaged region. In addition, dense (100) platelets with sizes ranging from 6 nm to 28 nm are observed all over the damage band. Some {111} platelets are also observed in the tail end of the damaged region. With increasing annealing temperature up to 973 K, in the Si with the oxide layer sample, the size and density of (100) platelets increase. A microcrack was formed in the damage band, as shown in Fig. 3(b). Very few {111} platelets are observed in the tail end of the damaged region. In the pure Si sample, the size of (100) platelets increases, while the density of (100) platelets decreases, as shown in Fig. 3(d). In order to investigate the dislocation loops in the damaged region, WBDF imaging of the damage band was performed. Fig. 4 presents frank loops in the damage band. The frank loops are visible in the weak-beam images as white dots. It can be seen that very dense small defect clusters and frank loops were formed in the damage band. The detail information of the density and size of frank loops is given in Fig. 5. Since the thickness of the thin foils was deduced from the number of fringes and considered statistical error, the uncertainly of the given densities is estimated above ±15%. In addition, the measurement error of frank loops is about 0.5 nm due to the resolution of TEM. After annealing at 773 K, in the Si with the oxide layer, the distribution of frank loops with sizes ranging from 2 nm to 18 nm is shown in Fig. 5(a). The mean size of frank loops is about 6 ± 0.5 nm and a number density is about 9.08 ± 1.45 × $10^{22}/m^3$. In the pure Si sample, Fig. 5(c) shows the frank loops with sizes ranging from 2 nm to 26 nm. The mean size of defect clusters is about 9.58 ± 0.5 nm and a number density is about 1.48 ± 0.29 × $10^{23}/m^3$. Dense frank loops are aligned parallel to the sample surface in the damage band. With increasing annealing temperature up to 973 K, in the Si with the oxide layer sample, Fig. 5(b) exhibits that the mean size of frank loops is about 9.61 ± 0.5 nm and a number density is about 1.45 ± 0.29 × $10^{23}/m^3$. The number density of frank loops increases with increasing temperatures from 773 K to 973 K. In the pure Si sample, the mean size of frank loops is about 7.04 ± 0.5 nm and a number density is about 1.18 ± 0.19 × $10^{23}/m^3$ [see Fig. 5(d)]. It indicates that the number density of frank loops decreases with increasing annealing temperature for the pure Si sample. The distribution of frank loops as a function of depth is shown in Fig. 6. The maximum number density of Frank loops is located at a depth of about 540 nm, which is independent with the samples. The bright-field images show that microcracks are located at a depth of 545 ± 10 nm. Therefore, a higher number density of frank loops is located near the region of microcracks.

3.3. Hydrogen related defect measurements

The evolution of hydrogen related defects with the annealing temperature studies by μ-RS is exhibited in Fig. 7. The spectra ranging from 1850 to 2250 cm^{-1} was investigated because the stretching modes of silicon–hydrogen (SiH_x) bonds, interstitial silicon–hydrogen (I_xH_y) bonds and vacancy–hydrogen (V_xH_y) bonds are included in the wavenumber range [13–15] ("I" denotes a Si interstitial and "V" denotes a Si vacancy). The background of spectra decays with increasing annealing temperature. A Raman band in the as-implanted samples was exhibited in the wavenumber ranging from 1900 to 2089 cm^{-1}, which is assigned to various superimposed multivacancy defects. Especially, a strong Raman line at 2025 cm^{-1}, can be attributed to the stretching mode of one hydrogen-terminated monovacancies (VH_1) bonds [13]. In addition, a strong Raman line at 2185 cm^{-1} is exhibited, which is attributed to the stretching mode of hydrogen saturated vacancy VH_3 bonds [13]. This defect has an additional stretching mode at 2158 cm^{-1}, which can be also seen in Fig. 7. The concentration of H related defects should be associated with the integrated intensity of their corresponding Raman peaks. Compared with two spectra in the as-implanted sample, the integrated intensity of the Raman band ranging from 1900 to 2089 cm^{-1} in the pure Si sample is increased by a factor of 1.4 as compared to the case of Si with the oxide layer. In parallel, in the spectra ranging of 2160–2200 cm^{-1} information about VH_3